ACHIRAL SUPERCRITICAL FLUID CHROMATOGRAPHY **SEPARATION OF HYDROBENZOIN**

APPLICATION NOTE

INTRODUCTION

Supercritical fluid chromatography (SFC) is a powerful separations technique that provides complementarity to normal phase high performance liquid chromatography (NP-HPLC). It offers a number of advantages to traditional separations techniques, including a low viscosity and highly diffusive mobile phase, making it amenable to fast flow analysis, as well as a diminished reliance on harsh organic solvents, requiring only small portions of modifiers like methanol or ethanol.

There are a number of stationary phases that have been developed for use with SFC, primarily for achiral separations. One such example is the 2-ethylpyridine (2-EP) bonded silica phase (SP). This SP typically offers good peak shape, especially for basic compounds, without the need for any additional additives. Although widely applicable for achiral separations, this SP shows no chiral recognition. This represents an area of need for a column(s) that could perform duel functionality (both achiral and chiral separations).

DAICEL's chiral stationary phases (CSPs) have shown considerable promise in this area. A number of examples have been shared previously, including the separations of steroids, caffeine and its analogs, methyl-substituted hippuric acids, and flavonoids. In this application, three isomers of hydrobenzoin (Figure 1) were successfully separated using CHIRALPAK® IA-3. The isomers of hydrobenzoin are used as chiral building blocks for a number of important synthetic applications, so being able to accurately and reliably quantitate sample purity is critical.

EXPERIMENTAL

A mixture containing (+)-hydrobenzoin, (-)-hydrobenzoin, and meso-hydrobenzoin was screened on DAICEL's library of immobilized chiral stationary phases (CSPs) under SFC conditions. The same mixture was also screened under SFC conditions using a 2-EP column of similar dimensions, utilizing a mobile phase of similar composition.

DISCUSSION

CHIRALPAK IA-3 (Figure 2) showed good initial selectivity under the screening conditions of 90:10 CO_2/MeOH, resulting in baseline separation of the (+)-hydrobenzoin, with partial coelution of the (-) and meso isomers. Further optimization to 92:8 CO_2/MeOH resulted in complete baseline separation of all three isomers.

In contrast, no such resolution could be found for 2-EP (Figure 3). The initial screening conditions of 90:10 CO_2/MeOH resulted in coelution of all three isomers. Further optimization to 95:5 CO_2/MeOH provided for a separation of the meso stereoisomer, however, and not surprisingly, no separation of the (+) and (-) enantiomers.

DAICEL

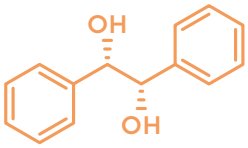
CHIRAL TECHNOLOGIES
DAICEL GROUP

WWW.CHIRALTECH.COM

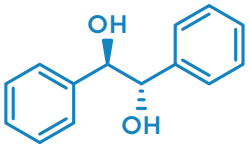
CHIRALCEL, CHIRALPAK and CROWNPAK are registered trademarks of DAICEL CORPORATION

FIGURE 1: THREE ISOMERS OF HYDROBENZOIN

(+)-HYDROBENZOIN



(-)-HYDROBENZOIN



MESO-HYDROBENZOIN

FIGURE 2: ACHIRAL SEPARATION OF HYDROBENZOIN ON IA-3

IA–3 COLUMN

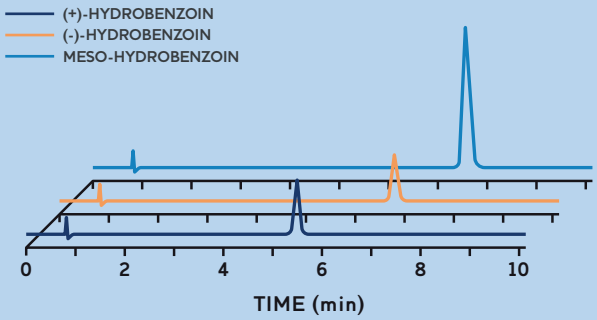
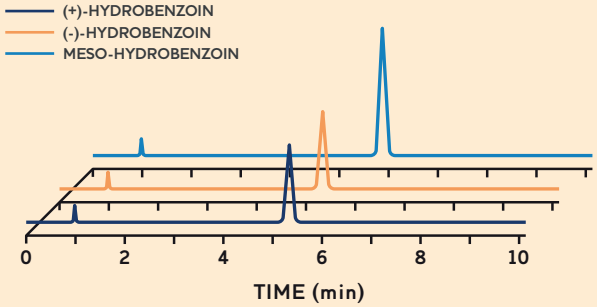


FIGURE 3: ACHIRAL SEPARATION OF HYDROBENZOIN ON 2-EP

2–EP COLUMN



CHROMATOGRAPHIC CONDITIONS FOR THE SEPARATION (+), (-), AND MESO-HYDROBENZOIN

Column: CHIRALPAK IA-3
Column Size: 4.6 mm i.d. x 150 mm long
Mobile Phase: CO_2/MeOH (92:8)
Flow rate: 4.0 mL/min.